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IS 13030 (1991): Method of test for laboratory determination of water content, porosity, density and related properties of rock material [CED 48: Rock Mechanics]

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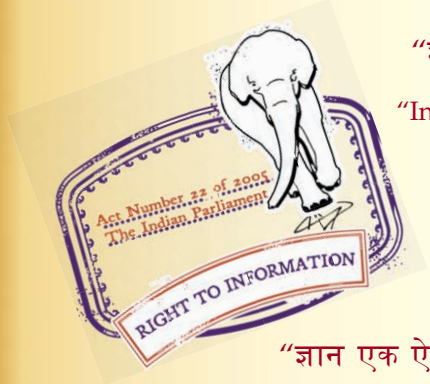

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Indian Standard

METHOD OF TEST FOR LABORATORY DETERMINATION OF WATER CONTENT, POROSITY, DENSITY AND RELATED PROPERTIES OF ROCK MATERIAL

UDC 624·121·43

BUREAU OF INDIAN STANDARDS
MANAK BHAVAN, 9 BAHADUR SHAH ZAFAR MARG
NEW DELHI 110002

May 1991

Price Group 3

FOREWORD

This Indian Standard was adopted by the Bureau of Indian Standards, after the draft finalized by the Rock Mechanics Sectional Committee had been approved by the Civil Engineering Division Council.

The presence of pores in fabric of a rock material decreases its strength and increases its deformability. A small volume fraction of pores can produce an appreciable mechanical effect.

Information on the porous nature of rock materials is frequently omitted from petrological descriptions but is required if these descriptions are to be used as a guide to mechanical performance. Sand stones and carbonate rocks in particular occur with a wide range of porosities and hence of mechanical character; igneous rocks that have been weakened by weathering processes also have typically high porosities.

Most rocks have similar grain densities and therefore have porosity and dry density values that are highly correlated. A low density rock is usually highly porous. It is often sufficient, therefore, to quote values for porosity alone but a complete description requires values for both porosity and density.

Different methods of determination of density and porosity described in the standard are suitable for different types of rocks and sample. While method using saturation and caliper technique and using saturation and buoyancy technique are suitable for regular and irregular shape of sample respectively of those rocks which do not appreciably swell or disintegrate when immersed in water, the method using mercury displacement and grain specific gravity technique is suitable for irregular shape of such rock which are liable to swell or disintegrate when immersed in water.

These methods are based on the suggested method of International Society of Rock Mechanics.

In reporting the results of a test or analysis made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS 2 : 1960 'Rules for rounding off numerical values (*revised*)'.

Indian Standard

METHOD OF TEST FOR LABORATORY DETERMINATION OF WATER CONTENT, POROSITY, DENSITY AND RELATED PROPERTIES OF ROCK MATERIAL

1 SCOPE

This standard gives the procedure for the test to evaluate water content, porosity, density and related properties of rock material. The following test methods have been covered:

a) Determination of the water content of rock sample;

b) Determination of porosity and density using saturation and caliper technique;

c) Determination of porosity and density using saturation and buoyancy; and

d) Determination of porosity and density using mercury displacement and grain specific gravity technique.

2 DEFINITIONS AND SYMBOLS

2.1 Symbols

For the purpose of this standard unless otherwise defined in the text, the following symbols shall apply:

M_s = Mass of grain (the solid component of the sample)

V_s = Volume of grain

M_w = Mass of pore water

V_w = Volume of pore water

P_w = Density of water

V_a = Volume of pore air

2.2 Definitions

For the purpose of this standard, physical properties pertinent to the methods of test shall be described as follows.

2.2.1 Bulk Sample Mass $M = M_s + M_w$

2.2.2 Bulk Sample Volume $V = V_s + V_v$

2.2.3 Pore (Voids) Volume $V_v = V_w + V_a$

2.2.4 Water Content, w

$$= \frac{M_w}{M_s} \times 100 \text{ (percent)}$$

2.2.5 Degree of Saturation, S_r

$$= \frac{V_w}{V_v} \times 100 \text{ (percent)}$$

2.2.6 Porosity $n = \frac{V_v}{V} \times 100$ (percent)

2.2.7 Void Ratio $e = \frac{V_v}{V_s}$

2.2.8 Density or Bulk Density or Mass Density

$$(\rho) = \frac{M}{V} = \frac{M_s + M_w}{V} \text{ kg/m}^3$$

2.2.9 Relative Density

$$\text{(mass specific gravity)} \; G_m = \frac{\rho}{\rho_w}$$

2.2.10 Dry density $\rho_d = \frac{M_s}{V}$ kg/m^3

2.2.11 Dry Relative Density

$$\text{(dry specific gravity)} \; G_d = \frac{\rho_d}{\rho_w}$$

2.2.12 Saturated Density

$$\rho_{sat} = \frac{M_s + V_v \cdot \rho_w}{V} \text{ kg/m}^3$$

2.2.13 Saturated Relative Density

$$\text{(saturated specific gravity)} \; G_{sat} = \frac{\rho_{sat}}{\rho_w}$$

2.2.14 Solid Density or Density of Rock Material $\rho_s = \frac{M_s}{V_s}$ kg/m^3

2.2.15 Solid Relative density

$$\text{(solid specific gravity)} \; G_s = \frac{\rho_s}{\rho_w}$$

2.2.16 Unit Weight $\gamma = \rho \times g$ N/m^3

3. TEST SAMPLE

A representative sample for testing should generally comprise several rock lumps, each in order of magnitude larger than the largest grain or pore size. Microfissures of similar size to that of a rock will cause erratic results, their presence should be noted and if possible the lump size increased or reduced to specifically include or exclude the influence of such fissures.

4 DETERMINATION OF THE WATER CONTENT OF A ROCK SAMPLE

4.1 Object

This method of test covers the procedure of determining the mass of water contained in a rock sample as a percentage of the oven dry sample mass.

4.2 Apparatus

a) An oven capable of maintaining a temperature of 105 ± 3°C for a period of at least 24 h.

b) A sample container non-corrodible material including an airtight lid.

c) A desiccator to hold sample container during cooling.

d) A balance of adequate capacity, capable of weighing to an accuracy of 0·01 percent of the sample mass.

4.3 Procedure

4.3.1 The container with its lid is cleaned and dried, and its mass m, is determined.

4.3.2 A representative sample comprised at least 10 lumps each having either a mass of at least 50 g or a minimum dimensions of ten times the maximum grain size, whichever is greater, is selected. For *in situ* water content determination sampling, storage and handling precautions should be such that water content remains within 1 percent of the *in situ* value.

4.3.3 The sample is placed in the container, the lid replaced and the mass m_2 of the sample plus container determined.

4.3.4 The lid is removed and the sample dried to constant mass at a temperature of 105 ± 3°C.

4.3.5 The lid is replaced and the sample allowed to cool in the desiccator for 30 minutes. The mass m_3 of sample plus container is determined.

4.4 Calculation and Reporting of Results

4.4.1 The water content shall be calculated from the following formula:

Water content, w

$$= \frac{\text{Pore water mass } M_w}{\text{Grain mass } M_s} \times 100 \text{ (percent)}$$

$$= \frac{m_2 - m_3}{m_3 - m_1} \times 100 \text{ (percent)}$$

m_1 = Mass in g of the container with its lid at room temperature

m_2 = Mass in g of the container with its lid and the sample at room temperature

m_3 = Mass in g of the container with its lid and the sample after drying

4.4.2 The water content should be reported to the nearest 0·1 percent stating whether this corresponds to *in situ* water content, in which case precautions taken to retain water during sampling and storage should be specified.

5 POROSITY AND DENSITY DETERMINATION USING SATURATION AND CALIPER TECHNIQUES

5.1 Object

5.1.1 This method of test covers the procedure for determining the porosity and dry density of rock samples in the form of specimen of regular geometry.

5.1.2 The method is applicable only to nonfriable coherent rocks that can be machined and do not appreciably swell or disintegrate when they are oven dried or are immersed in water. The method is suitable when regularly shaped specimens are required for other test purposes.

5.2 Apparatus

a) An oven capable of maintaining a temperature of 105 ± 3°C for a period of at least 24 h.

b) A desiccator to hold specimen during cooling.

c) A measuring instrument such as vernier or micrometer caliper, capable of reading specimen dimensions to an accuracy of 0·1 mm.

d) Vacuum saturation equipment such that the specimen can be immersed in water under a vacuum of less than 0·8 kPa for a period of at least one hour.

e) A sample container of non-corrodible material, including an airtight lid.

f) A balance of adequate capacity for determining the mass of a specimen to an accuracy of 0·01 percent of the specimen mass.

5.3 Procedure

5.3.1 Select at least three specimens from a representative sample of material. Machine each specimen to conform closely to the geometry of a right cylinder or prism. The minimum size of each specimen should either be such that its mass is at least 50 g (for an average density rock a cube with sides of 27 mm will have sufficient mass) or such that its minimum dimension is at least ten times the maximum grain size, whichever is the greater.

5.3.2 Repeat the following procedure for each of the specimen in the sample:

i) Determine the external dimension and then bulk volume V of each specimen with the vernier or calipers. Measurement should be accurate to 0·1 mm. An average of three separate measurement should be obtained for each dimension.

ii) Place the specimen in an oven and dry at 105 ± 3°C. For this test method specimens should be of sufficient coherence not to require containers, but these should be used if the rock is at all friable or fissible. The specimen is deemed to be dry when the difference between successive determinations of mass of the cooled specimen at intervals of 4 hours does not exceed 0·1 percent of the original mass of the specimen.

iii) Remove the specimen from the oven and place in a desiccator to cool.

iv) Determine the dry mass, M_s, of the specimen.

v) Immerse the specimen in a container of water and place the container with the specimen in a vacuum of less than 0·8 kPa for a period of one hour with periodic agitation to remove trapped air.

vi) Determine the water temperature, t, to the nearest degree centigrade.

vii) Remove the specimen from the container and surface dry it using a moist cloth. Care should be taken to remove only surface water and to ensure that no fragments are lost.

viii) Determine the saturation mass, M_{sat}, of the specimen.

5.4 Calculations

a) For each specimen calculate the pore volume, V_v, by the following formula:

$$V_v = \frac{M_{sat} - M_s}{\rho_w}$$

where ρ_w = density of water at temperature, t.

b) For each specimen calculate the bulk volume, V, from the external dimensions.

c) For each specimen calculate the dry density, ρ_d, by the following formula:

$$\rho_d = \frac{M_s}{V} \text{ (kg/m}^3\text{)}$$

d) For each specimen calculate the porosity, n, by the following formula:

$$n = \frac{V_v}{V} \times 100 \text{ (percent)}$$

e) Calculate average values of porosity and dry density for the sample.

5.5 Reporting of Results

a) Report the individual dry density and porosity values for each specimen in the sample, together with the average values of dry density and porosity for the sample. Density values should be given to the nearest 10 kg/m³ and porosity values to the nearest 0·1 percent.

b) Report that the bulk volume was obtained by measurement of dimension by caliper or vernier and the pore volume was obtained by water saturation.

c) Record any change in the shape and size of the rock specimen during wetting or drying.

d) Record the following general information:

i) Project title.

ii) Sampling technique and sample identifications number.

iii) Dates of sampling and of testing.

iv) Lithological description of the rock samples.

6 POROSITY AND DENSITY DETERMINATION USING SATURATION AND BUOYANCY TECHNIQUES

6.1 Object

This method of test covers the procedure for determining the porosity and the dry density of a rock sample in the form of lumps or aggregate of irregular shape-geometry. It may also be applied to a sample in the form of specimens of irregular geometry.

6.1.1 The method should only be used for rocks that do not appreciably swell or disintegrate when oven-dried and immersed in water.

6.2 Apparatus

a) An oven capable of maintaining a temperature of 105 ± 3°C for a period of at least 24 h.

b) A sample container of non-corrodible material, including an air-tight lid.

c) A desiccator to hold sample container during cooling.

d) A vacuum vessel such that the sample can be immersed in water under a vacuum of less than 0·8 kPa for a period of at least 1 h.

e) A balance of adequate capacity, capable of weighing to an accuracy of 0·01 percent of the sample weight.

f) An immersion bath and a wire basket or perforated container, such that the sample immersed in water can be freely suspended from the stirrup of the balance to determine the saturated-submerged weight. The basket should be suspended from the balance by a fine wire so that only the wire intersects the water surface in the immersion bath.

6.3 Procedure

a) A representative sample comprising at least 10 lumps of regular or irregular geometry,

each having either a mass of at least 50 g or a minimum dimension of at least 10 times the maximum grain size, whichever is the greater, is selected. The sample is washed in water to remove dust.

b) The sample is saturated by water immersion in a vacuum of less than 0·8 kPa for a period of at least 1 h, with periodic agitation to remove trapped air.

c) Determine the temperature, t, of the water in the immersion bath to the nearest degree centigrade.

d) Determine the mass, M_1, of the basket submerged in the immersion bath.

e) Transfer the sample under water to the basket in the immersion bath. Determine the saturated-submerged mass, M_2, of the basket plus sample to an accuracy of 0·01 percent of the sample mass.

f) Determine the mass, M_3, of a clean, dry sample container and lid.

g) Remove the sample from the immersion bath and surface dry the sample with a moist cloth, care being taken to remove only surface water and to ensure that no rock fragment are lost. Transfer the sample to the sample container and replace the lid. Determine the mass, M_4, of the saturated surface dry sample plus container.

h) Remove the lid and place the container with contents and lid in the oven and dry at 105 ± 3°C. The sample is deemed to be dry when the difference between successive determination of mass of the cooled sample at interval of 4 hours does not exceed 0·1 percent of the original mass of the sample.

j) Replace the lid, remove the container from the oven and place the whole in the desiccator to cool for 30 minutes.

k) Determine the dried mass, M_5, of the container with the oven dry sample.

6.4 Observations

Sample No. Date

Temperature of water, t, in Centigrade

Determination No.	(1)	(2)	(3)
1) Saturated-submerged mass of basket alone, M_1 in kg			
2) Saturated-submerged mass of basket + specimen, M_2 in kg			
3) Mass of container and lid, M_3 in kg			
4) Saturated surface dry mass of the sample plus container, M_4 in kg			
5) Dried mass of the container with sample, M_5 in kg			

6.5 Calculations

a) Calculate the saturated-submerged mass, M_{sub}, of the sample

$$M_{sub} = M_2 - M_1 \text{ (kg)}$$

b) Calculate the saturated-surface-dry mass, M_{sat}, of the sample

$$M_{sat} = M_4 - M_3 \text{ (kg)}$$

c) Calculate the dry mass (Grain Weight) M_s, of the sample

$$M_s = M_5 - M_3 \text{ (kg)}$$

d) Calculate the bulk-volume, V, of the sample by the following formula:

$$V = \left(\frac{M_{sat} - M_{sub}}{\rho_w} \right) \text{ (m}^3\text{)}$$

ρ_w = density of water at temperature t

e) Calculate the pore volume, V_v of the sample by following formula:

$$V_v = \left(\frac{M_{sat} - M_s}{\rho_w} \right) \text{ (m}^3\text{)}$$

f) Calculate porosity, n, of the rock sample by the following formula:

$$n = (V_v / V) \times 100 \text{ (percent)}$$

$$\text{or} = \left(\frac{M_{sat} - M_s}{M_{sat} - M_{sub}} \right) \times 100 \text{ (percent)}$$

g) Calculate the dry density, ρ_d, of the rock sample by the following formula:

$$\rho_d = \frac{M_s}{V} \text{ (kg/m}^3\text{)}$$

6.6 Reporting of Result

a) Report the porosity and dry density values or the sample to the nearest 0·1 percent and 10 kg/m³ respectively.

b) Report that the bulk volume was obtained by a buoyancy technique and that the pore volume was obtained by water saturation.

c) Report the following general information:

i) Project title

ii) Sampling technique and sample identification numbers

iii) Date of sampling and testing

iv) Lithological description of the rock samples.

7 POROSITY AND DENSITY DETERMINATION USING MERCURY DISPLACEMENT AND GRAIN SPECIFIC GRAVITY TECHNIQUES

7.1 Object

a) This method of test covers the procedure for determining the porosity and the density of a rock sample in the form of lumps or aggregate of irregular geometry.

b) This is particularly suitable if the rock material is liable to swell or disintegrate if immersed in water. The test is also applicable to regularly shaped rock specimens or to coherent rock materials but other techniques are usually found more convenient in these cases.

7.2 Apparatus

a) An oven capable of maintaining a temperature of 105 ± 3°C for a period of at least 24 h. It should have forced ventilation exhausting to outside atmosphere.

b) Specimen containers of non-corrodible material, including airtight lids.

c) A desiccator to hold specimen containers during cooling.

d) A balance of adequate capacity, capable of mass determination to 0·01 percent of sample mass.

e) A mercury-displacement volume measuring apparatus capable of measuring specimen volume to 0·5 percent.

f) Grinding equipment to reduce the sample to a pulverized powder less than 150 mm in grain size.

g) A calibrated volumetric flask and stopper (conveniently 50 cm^3).

h) A constant temperature water bath.

j) A vacuum apparatus capable of maintaining a vacuum with a pressure of less than 0·8 kPa.

k) A soft brush of camel hair or of similar softness.

7.3 Procedure

a) A representative sample is selected comprising at least ten rock lumps, the shape and size of lumps suiting the capabilities of the volume measuring apparatus. The minimum size of each lump should preferably be either such that its mass exceeds 50 g or such that its minimum dimension is at least 10 times the maximum grain size, whichever is the greater. Specimen and swelling or fissible rock should be sampled and stoned to retain water content to within 1 percent of its *in situ* value prior to testing.

b) Repeat the following procedure for each of the specimens in the sample:

i) Brush each specimen to remove loose material and measure its volume, V, by mercury displacement.

ii) Carefully remove mercury adhering to the sample, ensuring that no rock fragment are lost.

iii) Determine the mass, M_1, of a suitable clean, dry, container and its lid.

iv) Place the specimen in the container, replace the lid and determine the mass, M_2, of the container plus specimen at initial water content.

v) Remove the lid and the specimen is oven dried to constant mass at a temperature of 105 ± 3°C allowed to cool for 30 minutes in a desiccator. The mass, M_3, of container plus oven dry specimen is determined.

c) Crush all the rock specimens and grind to a grain size not exceeding 150 mm. A number of representative sub-samples of about 15 g of the pulverized material are selected and oven-dried.

d) Determine the mass, M_4, of a clean, dry volumetric flask plus stopper to an accuracy of 0·001 g.

e) Fill the flask with a liquid such as kerosene that is non-reactive with the rock.

f) Bring the flask to equilibrium temperature in the constant temperature bath and adjust the liquid level accurately to the 50 cm^3 graduation. Remove the flask from the constant temperature bath, insert the stopper and clean and dry the outside of the flask.

g) Determine the mass, M_5, of the flask and liquld to an accuracy of 0·01 percent of the total mass.

h) Empty and dry the flask and add the representative 15 g sub-sample of dry pulverized rock with the aid of a funnel.

j) Determine the mass, M_6, of flask, sample and stopper to an accuracy of 0·001 g.

k) The flask and sub-sample are evacuated for about 20 minutes and sufficient fluid added to thoroughly wet the sample. Further fluid is then added and the flask is carefully evacuated to remove air. The flask is replaced in the constant temperature water bath and the liquid level adjusted accurately to the 50 cm^3 graduation.

m) The stoppered flask with its contents is allowed to cool and its mass, G, is determined to 0·01 percent of the total mass.

n) Steps (e) to (m) are repeated for each subsample of pulverized material.

7.4 Precautions

a) It is preferable to use self-indicating silica gel as the desiccant.

b) Metallic mercury is a toxic material and should be handled with considerable care. It is principally absorbed via the respiratory tract as elemental mercury vapour but there is also some absorption through the gastro-intestinal tract. Spillages are difficult to clean up completely owing to the propensity of the substance, if left, to divide into miniscue globules which can generate unacceptably high levels of mercury vapour in the atmosphere of a room. Tests should be carried out in fume cupboard to avoid accumulation of vapour, and mercury spillages should be liberally sprinkled with powdered sulphur which takes up the metallic mercury to form the less volatile sulphide.

c) The density of some non-reactive liquids may vary appreciably. It is essential that a consistent source of liquid is used for each test and that volumes are adjusted in the constant temperature bath.

7.5 Observations

Sample No. Date

Specimen No.	1	2	3
1 Bulk volume of specimen by mercury displacement, V, in M^3			
2 Mass of dry container plus lid, M_1 in kg			
3 Mass of the container and sample, + lid, M_2 in kg			
4 Mass of the container and oven dried sample, + lid, M_3 in kg			
5 Mass of the dry flask (volumetric) and stopper, M_4 in kg			
6 Mass of volumetric flask plus liquid stopper, M_5 in kg			
7 Mass of flask, stopper and sample, M_6 in kg			
8 Mass of flask, sample liquid 4 stopper, M_7 in kg			
9 Wet mass of sample $= M_2 - M_1$ (kg)			
10 Dry mass of sample $M_3 = M_3 - M_1$ (kg)			
11 Mass of water in the sample $= M_2 - M_3$ (kg)			
12 Moisture content $w = \frac{M_2 - M_3}{M_2 - M_3} \times 100$ (percent)			

7.6 Calculations

a) Calculate the grain density, ρ_s, of the pulverized rock material by the following formula:

$$\rho_s = \frac{M_6 - M_4}{V_f \left[1 - \left(\frac{M_7 - M_6}{M_5 - M_4} \right) \right]} \text{ (kg/m}^3\text{)}$$

where

V_f = Calibrated volume, in m^3 of the volumetric flask.

b) For each specimen in the sample calculate the following:

i) Dry density, ρ_d, by the following formula:

$$\rho_d = \frac{M_3}{V}$$

ii) Porosity, n, by the following formula:

$$n = \left[\frac{\rho_s - \rho_d}{\rho_s} \right] \times 100 \text{ (percent)}$$

c) From the values for the individual specimens, calculate the average dry density, porosity, moisture content and grain density for the sample.

7.7 Reporting of Results

a) Report the individual dry density, porosity and moisture content values for each specimen in the sample together with the average values of dry density, porosity and moisture content for the sample. Density values shall be given to the nearest 10 kg/m^3, porosity values to the nearest 0·1 percent and moisture content values to the nearest 0 1 percent.

b) Report that the bulk volume was obtained using a mercury displacement technique, and that the porosity was calculated from grain volume measurement using a pulverization technique.

c) Record any gross change in the shape or competence of the rock specimens during drying.

d) Record the following general information:

i) Project title

ii) Sampling technique and sample identification number

iii) Dates of sampling and of testing

iv) Lithological description of the rock sample.

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